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J.J.-W. Lee, Y. Wang,
I.K. Lloyd, and B.R. Lawn[1]*

Department of Materials Science and Engineering, University of Maryland, College Park, MD 20742-2115, USA; and [1]Materials Science and Engineering Laboratory, National Institute of Standards and Technology, 100 Bureau Drive, Bldg. 223, Gaithersburg, MD 20899-8520, USA; *corresponding author, brian.lawn@nist.gov

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Joining Veneers to Ceramic Cores and Dentition with Adhesive Interlayers

ABSTRACT

Adhesive joining of veneers to cores offers potential simplicity and economy in the fabrication of all-ceramic crowns. We tested the hypothesis that resin-based adhesives can be used for such fabrication without compromising mechanical integrity of the crown structure. A simple test procedure for quantifying this hypothesis was proposed. A model glass veneer layer 1 mm thick (representative of porcelain), adhesively bonded onto a glass-like core substrate (ceramic or dental enamel), was loaded at its top surface with a hard sphere (occlusal force) until a radial crack initiated at the veneer undersurface. The critical loads for fracture, visually observable in the transparent glass, afforded a measure of the predisposition for the adhesive to cause veneer failure in an occlusal overload. Two adhesives were tested, one a commercial epoxy resin and the other a relatively stiff in-house-developed composite. The results confirmed that stiffer adhesives provide higher resistance to failure.

KEY WORDS: adhesive joining, glass, contact loading, veneer failure.

INTRODUCTION

All-ceramic dental crowns generally consist of an aesthetic but weak porcelain veneer layer fused at elevated temperature to a functional and strong core ceramic, with the porcelain applied layer by layer by a dental technician (McLean, 1979, 1983). The process of joining is time- and labor-intensive, and is subject to potentially deleterious residual stresses from mismatch in coefficients of thermal expansion (CTE). An attractive alternative is to fabricate veneer and core individually by an alternative manufacturing route, and then to bond the layers together chemically at low temperatures by means of a polymeric resin-based adhesive. This route avoids CTE stresses (although perhaps not shrinkage stresses), and establishes a soft interfacial barrier for arresting cracks formed in any one layer (Clegg *et al.*, 1990). Onlays and inlays are joined onto prepared dentition by an analogous cementation procedure. The use of resin-based adhesives has its own possible drawbacks, most obviously the introduction of a weak interface, rendering the system liable to delamination and spalling. Perhaps more important, because the interface is relatively compliant, the prospect arises for flexure during occlusal loading and consequent failure of the veneer by crack initiation from a flaw at the bottom surface (Chai and Lawn, 2000). Once initiated, such cracks spread radially outward from the source into an elongate elliptical configuration—hence the term "radial" crack—and eventually traverse the veneer to the top surface.

The challenge, then, is to find and test adhesives that are both stiff enough to minimize veneer flexure and strong enough to resist delamination. The present study pursues this challenge by the use of a model layer system (Fig. 1) (Chai and Lawn, 2000), consisting of a glass veneer plate of thickness d = 1.0 mm joined to a much thicker glass substrate plate by either of 2 resin-based adhesives—one a standard epoxy of Young's modulus 2.3 GPa, and the other an in-house-processed particle-filled composite of modulus 20.4 GPa—of prescribed thickness h. Because of monomer ingredients, these 2 adhesives bond strongly to silanized or primed ceramics. They also encompass the modulus values of traditional dental cements, which range from 4 GPa to 12 GPa (Kim *et al.*, 2003). The resulting layer structure is loaded at its top surface with a spherical indenter, thus placing the veneer in flexure and inducing radial cracking at the bottom surface. Such a system captures the essence of an occlusally loaded, adhesive-bonded veneer/core crown structure, at the same time facilitating direct visualization of the fracture modes in the veneer layer from the side and from below. This allowed for quantitative testing of our hypothesis that resin-based adhesives can be used for joining veneers to cores without compromising mechanical integrity of the crown structure.

MATERIALS & METHODS

Glass plates of thickness d = 1.0 mm (veneer) and 12.5 mm (base) were used as adjoining brittle layers. The thin plates were abraded at their bottom surfaces

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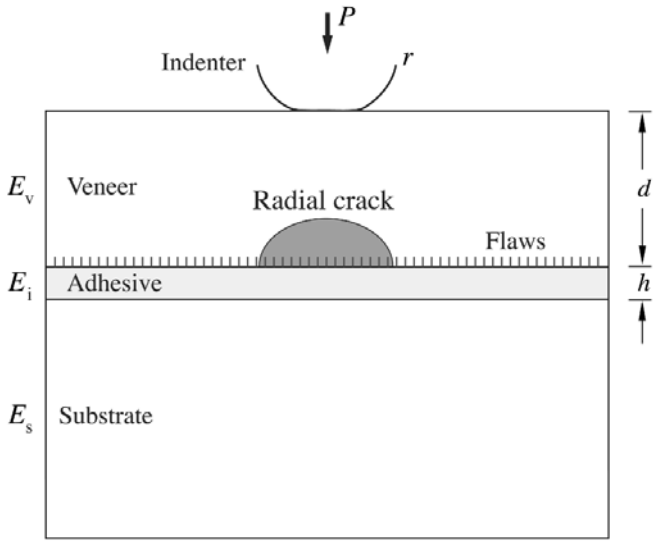

Figure 1. Schematic of glass/adhesive/glass layer system in contact loading, showing key variables.

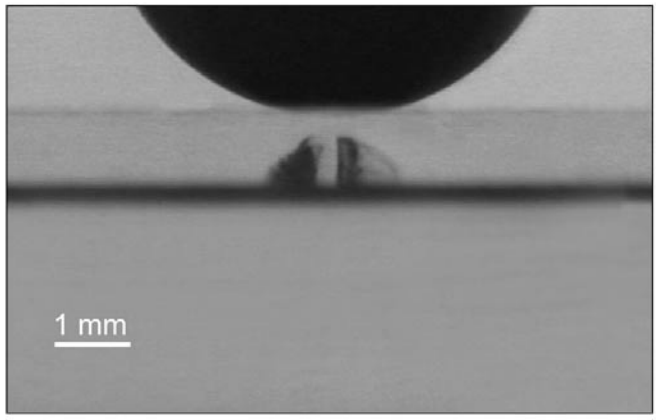

Figure 2. Photograph showing contact-induced radial cracks at bottom surface of glass veneer layer of thickness d = 1.0 mm bonded to glass substrate with composite adhesive of thickness h = 100 μm, at initiation load P = 600 N. Side view, crack visible as elliptical shadow.

with 600 SiC grit (Chai *et al.*, 1999). This treatment provided starting flaws of controlled size for the ensuing radial fracture initiation, equivalent to the sandblasting treatments used by dental clinicians, thereby producing a worst-case scenario for veneer strength properties (Zhang *et al.*, 2006). The same plates were etched (10% hydrofluoric acid, 30 sec) at their top surfaces for removal of surface handling flaws, thus precluding spurious cone fractures during testing (Bhowmick *et al.*, 2005). Side walls were polished to allow for direct observation of crack evolution.

The veneer and base glass plates were joined by 2 different adhesives. An epoxy resin (Harcos Chemicals, Bellesville, NJ, USA) was used as a simple adhesive with good bonding, well-documented in studies of this kind (Chai *et al.*, 1999). We produced a composite with uncommonly high modulus by loading 72 mass% spherical alumina particles (NanoTek, Nanophase Technologies Corp., Romeoville, IL, USA) of mean diameter 45 nm into a monomer blend of 50 mass% bisphenol-A-glycidyldimethacrylate (bis-GMA, Esstech, Essington, PA, USA) and 50 mass% triethylene glycol dimethacrylate (TEDGMA, Esstech, Essington, PA, USA). The mixtures were stirred for 4 hrs to break up any agglomeration of particles and to produce a uniform microstructure, as confirmed by scanning electron microscopy of sections (Wang *et al.*, 2007).

Opposing surfaces of the glass plates were first silanized (3M ESPE RelyX Ceramic Primer, St. Paul, MN, USA) and then joined with the 2 adhesives in their as-mixed forms. Epoxy joins were formed and cured at room temperature for one day. Composite joins were formed at room temperature and heated in an oven at 120°C for at least 6 hrs, to enable the organic matrix to crosslink. The resulting composite was relatively uniform, with alumina filler dispersed throughout the matrix. We obtained adhesive interlayers of prescribed thicknesses h by squeezing the opposing glass plates tight under light pressure (small h) or by inserting spacers between the layers (large h). Adhesive interlayer thicknesses were measured at specimen sections by a micrometer, producing a working range h = 65 μm to 650 μm. This covers the range of dental cement thicknesses used in adhering crowns to tooth structure (McLean, 1979). Elastic moduli of the adhesives were measured at the same sections for different values of h by means of a nanoindenter (Oliver and Pharr, 1992), with contact diameter of *ca.* 50 μm (*i.e.*, much greater than the 45-nm particle scale) (Wang *et al.*, 2007). Means and standard deviations were E_i = 2.3 ± 0.1 GPa for epoxy and E_i = 20.4 ± 0.6 GPa for the filled composite.

Contact with a WC spherical indenter of radius r = 3.18 mm, mounted into a mechanical testing machine, was applied at the specimen top surface (Chai and Lawn, 2000; Bhowmick *et al.*, 2005; Zhang *et al.*, 2005; Hermann *et al.*, 2006). Loading was increased monotonically until radial cracks initiated at the bottom surface of the veneer plate (Fig. 1). A video camera system was used to view the crack development, from both the side through the veneer plate wall and below through the base.

Variations in the measurement of experimental variables—load P, layer thickness d, and sphere radius r—were less than 1%, so that uncertainties in the ensuing data could be largely ascribed to material variation.

RESULTS

A side view of a glass/glass specimen bonded with composite adhesive of thickness h = 100 μm and contact-loaded at P_R = 600 N revealed a typical radial crack pattern in the veneer layer (Fig. 2). Upon further loading, the crack arms expanded sideways and upward, ultimately penetrating through to the top surface. Views from below the base confirmed the incidence of several such radial cracks, at more or less equal angles, forming a characteristic "star" pattern (Chai *et al.*, 1999). The side view in Fig. 2 highlights one such radial crack, approximately normal to the observation direction. In our experiments, no subsidiary fracture modes, *e.g.*, interfacial delamination or top-surface cone cracking, were observed in any specimens up to loads P = 1200 N.

Load P_R to produce veneer radial cracks was plotted as a function of adhesive thickness h, for epoxy and filled-composite adhesives (Fig. 3). A logarithmic scale was used simply to show the curve shifts for the different adhesives with greater clarity. The data points with error bars are means and standard deviations of 6 tests for each adhesive at one clinically representative thickness, h = 150 μm; the other data points are individual test results in specimens of various adhesive

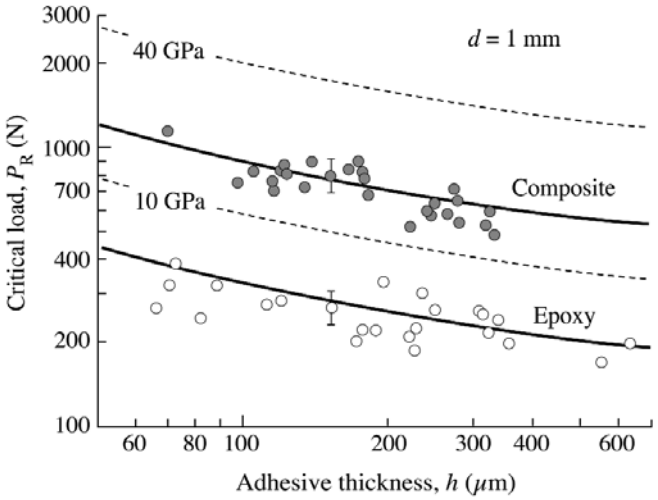

Figure 3. Plot of crack initiation load P_R *vs.* interlayer thickness h in logarithmic coordinates for glass veneer of thickness d = 1.0 mm bonded to glass substrate with adhesive of modulus E_i = 2.3 GPa (epoxy) and 20.4 GPa (composite). Data points with error bars at interlayer thickness h = 150 μm are means and standard deviations for minimum of 6 specimens each. Other data points are individual test results. Number of specimens n = 60. Solid lines are fits from Eq. 1. Dashed lines are for hypothetical adhesives of modulus E_i = 10 GPa and 40 GPa.

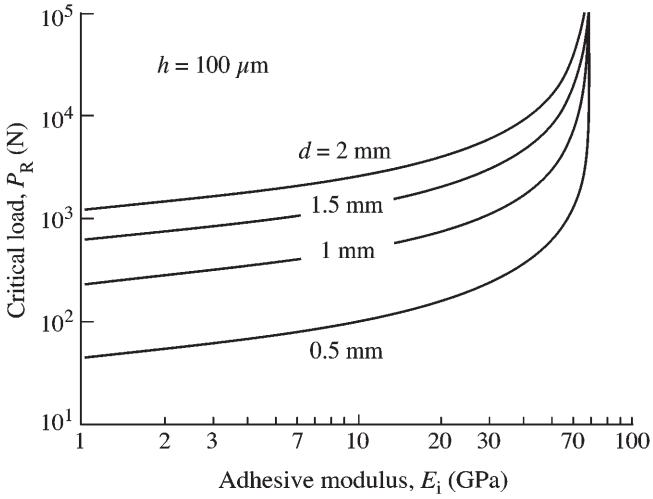

Figure 4. Calculated values of crack initiation load P_R as function of adhesive modulus E_i in logarithmic coordinates for all-glass veneer/substrate system bonded with adhesive of interlayer thickness h = 100 μm. Curves shown for selected veneer thicknesses d indicated.

thicknesses. Of primary interest here is a distinct upward shift in the data, by more than a factor of 3, from epoxy to filled-composite adhesive. A standard t test analysis of the datasets at h = 150 μm yields $p < 0.000002$, so that this shift may be considered significant. Note also that each dataset indicates monotonically decreasing P_R with increasing h, amounting to about a factor of 2 decline over the order-of-magnitude range covered. Thus, any decrease in fracture resistance from increase in adhesive thickness can easily be outweighed by a change to a stiffer adhesive.

The data trends (Fig. 3) can be quantified by an existing set of equations for the initiation of radial cracks in a veneer plate of modulus E_v and thickness d adhesively bonded to a thick substrate of modulus E_s (Kim *et al.*, 2003):

$$P_R = BSd^2/\log(E_v/E^*) \quad (1)$$

where B = 2 is a constant, S is the strength of the glass, and where we define an effective modulus E^* of the combined adhesive/substrate underlayer

$$E^* = E_i(E_s/E_i)^L \quad (2)$$

and where L is an exponent dependent on adhesive thickness

$$L = \exp\{-[\alpha + \beta \log(h/d)]^\gamma\} \quad (3)$$

with α = 1.18, β = 0.33, and γ = 3.13. These equations are based on an analysis of flexure of a contact-loaded brittle plate (veneer) on a compliant interlayer (adhesive), and assume that radial fracture occurs when the maximum tensile stress at the bottom surface of the plate reaches the strength S of the material (Chai *et al.*, 1999). The equations have been validated in independent experiments over a much wider range of adhesive thicknesses (from 5 μm to 2 mm) than covered here (Kim *et al.*, 2003). The solid curves through the data are predictions made from these equations by inserting: S = 130 MPa, E_v = 70 GPa, and d = 1.0 mm for the glass veneer; E_s = 70 GPa for the glass substrate; E_i = 2.3 GPa for the epoxy, and E_i = 20.4 GPa for the filled composite adhesive. It is seen that the solid curves fit the corresponding data trends within the scatter. Also included (as the dashed curves) are comparative predictions for E_i = 10 GPa (*i.e.*, in the vicinity of most dental cements) and for E_i = 40 GPa (composites containing ultra-high modulus fillers, *e.g.*, diamond). Again, the advantages of stiffer adhesives are apparent.

The formulation in Eqs. 1-3 establishes a basis for considering the role of geometric and material variables in veneer failure. As an illustrative example, retaining the same material parameters for glass, plots are shown for P_R as a function of adhesive modulus E_i, using logarithmic coordinates, for fixed adhesive thickness h = 100 μm and selected values of veneer thickness d (Fig. 4). The virtue of a high value of E_i is apparent. Also apparent is a strong dependence of P_R on d, a sensitivity that has been well-documented in the literature (Lawn *et al.*, 2001; Rhee *et al.*, 2001; Deng *et al.*, 2002).

DISCUSSION

We have used a simple contact loading test to quantify the suitability of resin-based adhesives as a means for joining veneers to a base core layer. The test is simple, making use of transparent glass plates in a model layer system for *in situ* detection of veneer radial cracking. The critical loads P_R to initiate such cracks provide a quantitative measure of resistance to veneer failure. In the clinical context, it is necessary to maintain $P_R >> 100$ N, *i.e.*, above the range of typical occlusal biting forces (McLean, 1979; Kelly, 1997, 1999). This condition is accomplished by both test adhesives, over a broad range of join thicknesses h, with an especially comfortable safety margin for the stiff composite. Increasing E_i above 20 GPa overcame any degradation from excessive adhesive thickness. The test

also verified the bonding capacity of the adhesive. Recall that, in our tests, no debonding was observed up to P_R = 1200 N. Contrast this to results from an earlier study on some commercial dental cements, where the layers delaminated catastrophically at low loads, indicating a total unsuitability of those cements for veneer/core joining (Kim *et al.*, 2003).

The present results therefore suggest that resin-based adhesives may provide a convenient means of joining brittle veneers to underlying ceramic cores (crowns) or tooth enamel (onlays and inlays). Potential advantages include: avoidance of CTE stresses (although care may be needed to avoid shrinkage stresses during polymeric curing); and arrest of veneer (or core) cracks at the soft/tough interlayer, thereby inhibiting spread of cracks from one layer to another. The adhesive needs to meet certain requirements:

(i) It should be stiff enough to minimize veneer flexure in occlusal loading, to optimize resistance to radial crack initiation. Part of our program is committed to developing such stiff resin-based adhesives for this purpose. However, there is a need for caution, because stiffer adhesives tend to be more brittle and could compromise the crack-arrest capacity, leading to catastrophic failure of the entire crown/tooth structure.

(ii) The adhesive should form a strong chemical bond with the adjoining surfaces. A rule of thumb is that the interfacial toughness should be at least one-half that of the bulk adhesive material, to avoid crack deflection and delamination (He and Hutchinson, 1989; Kim *et al.*, 2006). For the adhesives used here, inclusion of a monomer component in the adhesive appears to meet the need.

We have demonstrated how Eqs. 1-3 can be used to elucidate the dependence of critical load P_R for veneer fracture on adhesive modulus E_i and veneer thickness d. We may further note the appearance of strength S in Eq. 1, which suggests that control of the surface flaw state of the veneer is an important factor in veneer preparation (Zhang *et al.*, 2006). Dummy tests on as-polished glass veneers show critical loads considerably higher than those with surface abrasion, it is reported here (Chai *et al.*, 1999). Last, sphere radius r is a relatively unimportant parameter in our tests, because radial crack initiation occurs at the veneer undersurface remote from the contact zone—necessary only to ensure that r does not become too small, to avoid spurious cone cracking at the intensified Hertzian contact.

While it is experimentally expedient to construct the test layer system entirely from glass, for the reasons outlined in the INTRODUCTION, Eq. 2 may, in principle, be expanded to cover the case of dissimilar veneer and substrate materials (Kim *et al.*, 2003). However, the formulation in Eq. 2 tends to overestimate E^* in the region of high E_s, so that veneer failures remain a threat with even the stiffest core ceramics. An all-glass system usefully provides a lower bound to P_R values.

Finally, data such as those presented here may serve one more useful purpose, in cases where it becomes difficult to measure E_i of ultra-thin interlayers, and where it is suspected that E_i might differ from the modulus of the bulk material (*e.g.*, by redistribution of the particulate density during joining). Given knowledge of the glass properties, along with the adhesive thickness, one may, in principle, 'deconvolute' the modulus E_i numerically from the data by using Eqs. 1-3.

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Certain equipment, instruments, or materials are identified in this paper to specify experimental details adequately. Such identification does not imply recommendation by the National Institute of Standards and Technology, nor does it imply that the materials are necessarily the best available for the purpose.

REFERENCES

Bhowmick S, Zhang Y, Lawn BR (2005). Competing fracture modes in brittle materials subject to concentrated cyclic loading in liquid environments: bilayer structures. *J Mater Res* 20:2792-2800.

Chai H, Lawn BR (2000). Role of adhesive interlayer in transverse fracture of brittle layer structures. *J Mater Res* 15:1017-1024.

Chai H, Lawn BR, Wuttiphan S (1999). Fracture modes in brittle coatings with large interlayer modulus mismatch. *J Mater Res* 14:3805-3817.

Clegg WJ, Kendall K, Alford NM, Button TW, Birchall JD (1990). A simple way to make tough ceramics. *Nature* 347:455-457.

Deng Y, Lawn BR, Lloyd IK (2002). Characterization of damage modes in dental ceramic bilayer structures. *J Biomed Mater Res* 63(B):137-145.

He MY, Hutchinson JW (1989). Crack deflection at an interface between dissimilar elastic materials. *Int J Solids Struct* 25:1053-1067.

Hermann I, Bhowmick S, Zhang Y, Lawn BR (2006). Competing fracture modes in brittle materials subject to concentrated cyclic loading in liquid environments: trilayer structures. *J Mater Res* 21:512-521.

Kelly JR (1997). Ceramics in restorative and prosthetic dentistry. *Ann Rev Mater Sci* 27:443-468.

Kelly JR (1999). Clinically relevant approach to failure testing of all-ceramic restorations. *J Prosthet Dent* 81:652-661.

Kim JH, Miranda P, Kim DK, Lawn BR (2003). Effect of an adhesive interlayer on the fracture of a brittle coating on a supporting substrate. *J Mater Res* 18:222-227.

Kim JW, Bhowmick S, Hermann I, Lawn BR (2006). Transverse fracture of brittle layers: relevance to failure of all-ceramic dental crowns. *J Biomed Mater Res B Appl Biomater* 79:58-65.

Lawn BR, Deng Y, Thompson VP (2001). Use of contact testing in the characterization and design of all-ceramic crown-like layer structures: a review. *J Prosthet Dent* 86:495-510.

McLean JW (1979). The science and art of dental ceramics. Vol. 1: The nature of dental ceramics and their clinical use. Chicago: Quintessence.

McLean JW (1983). The future for dental porcelain. In: Dental ceramics: proceedings of the First International Symposium on Ceramics. McLean JW, editor. Chicago: Quintessence Publishing Co.

Oliver WC, Pharr GM (1992). An improved technique for determining hardness and elastic modulus using load and displacement sensing indentation experiments. *J Mater Res* 7:1564-1583.

Rhee Y-W, Kim H-W, Deng Y, Lawn BR (2001). Contact-induced damage in ceramic coatings on compliant substrates: fracture mechanics and design. *J Am Ceram Soc* 84:1066-1072.

Wang J, Lee JJ, Lloyd IK, Wilson OC, Rosenblum M, Thompson VP (2007). High modulus nanopowder reinforced dimethacrylate matrix composites for dental cement applications. *J Biomed Mater Res A* (DOI:10.1002/jbm.a.31029).

Zhang Y, Bhowmick S, Lawn BR (2005). Competing fracture modes in brittle materials subject to concentrated cyclic loading in liquid environments: monoliths. *J Mater Res* 20:2021-2029.

Zhang Y, Lawn BR, Malament KA, Thompson VP, Rekow ED (2006). Damage accumulation and fatigue life of particle-abraded dental ceramics. *Int J Prosthodont* 19:442-448.